TABLE II (cont.)

Materials	Tensile Properties			Notched Izod Impact.	HDT	Shore D
	Yield PSI	Ultimate PSI	% Elong	Ft-Lb/In	Deg C 66 PSI	
HDPE/PET 40/60 Pulv		4100	5	0.7	68	70
HDPE/LDPE/PET 30/30/40 Flake		2010	4	0.4	62	64
HDPE/LDPE/PET 30/30/40 Pulv		2520	5	0.3	62	65
HDPE/LDPE/PET 40/30/30 Flake		1880	4	0.3	63	64
HDPE/LDPE/PET 40/30/30 Pulv		2450	5	0.2	63	65
HDPE/LDPE/PET 60/10/30 Flake		2330	4	0.3	67	66
HDPE/LDPE/PET 60/10/30 Pulv		3020	5	0.2	67	68
HDPE/LDPE 40/60 Flake		2530	17	0.5	47	59
HDPE/LDPE 40/60--No heat pulv	2500		90	0.6	51	60
HDPE/LDPE 60/40 Flake		2840	13	0.4	57	62
HDPE/LDPE 60/40 Pulv		2860	13	0.5	57	63
PP/PS 70/30 Flake	5320		16	0.6	93	76
PP/PS 70/30 Pulv	5120		10	0.4	90	76
PP/PS 70/30--No heat pulv		5200	8	0.4	91	75
PP/PS 30/70 Flake		5880	5	0.6	90	79
PP/PS 30/70 Pulv		5780	4	0.45	88	79
PP/PS 30/70--No heat pulv		5950	5	0.4	90	80
HDPE/LDPE/PP/PET 40/30/10/20 Flake		2300	6	0.2	59	65

TABLE II (cont.)

Materials	Tensile Properties			Notched Izod Impact	HDT	Shore D
	Yield PSI	Ultimate PSI	% Elong	Ft-Lb/In	Deg C 66 PSI	
HDPE/LDPE/PP/PET 40/30/10/20 Pulv	2710		10	0.3	59	65
HDPE/LDPE/PP/PET/ PS 40/30/5/20/5 Flake		2020	4	0.2	63	64
HDPE/LDPE/PP/PET/ PS 40/30/5/20/5 Pulv		2610	8	0.2	64	64
HDPE/LDPE/PP/PVC 55/30/10/5 Flake		2540	7	0.3	55	63
HDPE/LDPE/PP/PVC 55/30/10/5 Pulv		2390	6	0.3	65	63
HDPE/LDPE/PP/PET/ PVC 40/30/5/20/5 Pulv		2290	5	0.2	65	65
HDPE/LDPE/PP/PET/ PS/PVC 40/30/5/15/5/5 Pulv		2210	5	0.2	61	65

It is noteworthy that the tensile strengths of injection molded specimens made from pulverized ternary HDPE/LDPE/PP and HDPE/LDPE/PET powder of the invention are consistently higher than the tensile strengths of molded specimens molded from the as-received blended flake feedstocks of the same materials. This increase in tensile strength for the pulverized powder molded specimens indicates an increased compatibility of the polymer components in the blend.

It is further noteworthy that even though some of the scrap feedstocks set forth in the Examples comprised a plurality of polyolefins (e.g. HDPE, LDPE, PP) that are mutually thermodynamically incompatible, the feedstocks were nevertheless successfully injection molded, and the molded specimens did not exhibit delamination upon breaking in the mechanical property tests, indicating that chemical change occurred during pulverization and the polymers have been in-situ compatibilized during the solid state shear pulverization process of the invention.

Furthermore, as mentioned above, the pulverized recycled powders of the invention exhibited enhanced reactivity as compared to the flake feedstock M prior to pulverization. To facilitate studies of the chemical state (reactivity) of the pulverized powder, small samples (2-3 grams) of powder were collected from the discharge end of the extruder barrel.

Powder samples were loaded into quartz tubes for electron spin resonance (ESR) measurements. ESR spectra were acquired at room temperature on a modified continuous wave Varian E-4 spectrometer operating in the X-band (microwave frequency near 9 GHz).

Initial ESR studies were made of unpulverized flake LDPE and pulverized LDPE powder of the invention pulverized from the flake in accordance with the invention. It appears that reactive sites (free

radicals) are formed by polymer bond rupture during the solid state shear pulverization process and have lifetimes that can be estimated as several hours at ambient conditions. In general, ESR spectra of unpulverized feedstock flakes and pulverized powder stored at ambient conditions indicate the presence of stable peroxy radicals in both the LDPE, PP, and 70%HDPE/30%PP flakes and powder. However, the pulverized powders have greater free radical densities than the as-received (unpulverized) flakes in accordance with the invention, as shown in Figures 9, 10 and 11.

In Figure 11, a simulated ESR spectrum for unpulverized mixture of 70% HDPE/30% PP at room temperature is shown at the top. At the bottom, a simulated ESR spectrum of an incompatible blend of pulverized 70% HDPE/30% PP superimposed over an actual measured ESR spectrum of these pulverized blends are shown at the bottom. All spectra were normalized to the same mass. The simulated spectrum is determined by adding spectra of the individual blend components, each scaled according to their fraction in the blend. Because the actual spectrum is larger (more intense) than the simulated one, it implies an interaction between HDPE and PP during the pulverization process.

Furthermore, differential scanning calorimetry (DSC) using a Perkin-Elmer DSC-7 unit provided unambiguous evidence that solid state shear pulverization in accordance with the invention produces significant chemical changes suggesting a compatibilizing effect. Dramatic changes in the melting and crystallization peaks of HDPE/LDPE and HDPE/PP blends were observed after pulverization of dry blends of these incompatible polyolefins, as shown in Figures 12-18.

Table III summarizes the DSC results. In Table III, F adjacent the material refers to post-consumer unpulverized flake or chunk material and Pul refers to pulverized material.

TABLE III

Melting							Crystallization		
Thermal History: Original									
Material	Sample ID	Mass (mg)	Desc.	Onset Tm(°C)	Peak Tm(°C)	Delta H (J/g)	Onset Tc(°C)	Peak Tc(°C)	Delta H(J/g)
LDPE-F				122.269	126.828	78.239	114.402	111.047	-106.103
LDPE-Pul				125.187	129.128	75.209	117.334	115.295	-108.787
HDPE-F				123.944	131.527	152.512	117.693	114.271	-159.981
HDPE-Pul				124.816	128.569	158.309	116.733	114.948	-162.713
PP-F				153.063	163.379	76.431	127.389	123.691	-93.975
PP-Pul				152.376	162.793	68.787	122.751	120.058	-89.513
PP-Pul				156.863	168.022	76.919	124.834	121.69	-81.76
Thermal History: Cooled at 10°C/min.									
LDPE-F				122.759	126.828	78.239			
LDPE-Pul				124.392	128.161	90.659			
HDPE-F				123.679	131.895	190.123			
HDPE-Pul				121.573	127.777	156.829			
PP-F				153.852	162.61	83.58			
PP-Pul				149.058*	160.175*	76.933			
PP-Pul				159.196*	164.461*	63.727			

* averaged over more than one peak

TABLE III(Cont.)

Thermal History: Original							
Material (Composition)	Peak	Onset Tm(°C)	Peak Tm(°C)	Delta H (J/g)	Onset Tc(°C)	Peak Tc(°C)	Delta H(J/g)
HDPE/PP (70/30)-F	HDPE	123.426	130.08	170.703	116.125	114.185	-140.942
	PP	152.397	162.927	71.215	129.05	125.37	- 49.51
HDPE/PP (70/30)-Pul	HDPE	121.779	128.673	174.066	116.975	114.847	-158.279
	PP	151.299	162.454	75.882	125.387	122.342	- 81.961
HDPE/LDPE/PP(60/30/10)-F	HDPE/LDPE	124.153	134.265	154.237	118.401	115.666	-179.678
	PP	156.104	163.736	49.94	No PP crystallization peak observed		
HDPE/LDPE/PP(60/30/10)-Pul	HDPE/LDPE	123.399	129.199	169.264	118.659	116.957	-134.321
	PP	162.287	167.359	-109.55	127.829	124.952	-74.87
HDPE/LDPE(40/60)-F	HDPE/LDPE	122.419	127.927	108.524	116.42	114.586	-123.243
HDPE/LDPE(40/60)-Pul	HDPE/LDPE	121.257	128.404	114.058	117.351	115.394	-115.204
HDPE/LDPE(60/40)-F	HDPE/LDPE	124.538	131.64	123.379	116.939	114.382	-132.586
HDPE/LDPE(60/40)-Pul	HDPE/LDPE	122.687	127.89	110.815	117.671	115.784	-107.01

TABLE III(Cont.)

Thermal History: Cooled at 10°C/min.							
HDPE/PP(70/30)-F	HDPE	122.184	128.006	183.304			
	PP	149.671	160.991	68.5			
HDPE/PP(70/30)-Pul	HDPE	121.188	128.182	188.188			
	PP	155.211	160.277	77.159			
HDPE/LDPE/PP(60/30/10)-F	HDPE/LDPE	124.856	132.305	165.523			
	PP	155.903	161.912	70.05			
HDPE/LDPE/PP(60/30/10)-Pul	HDPE/LDPE	123.706	128.93	154.731			
	PP	158.317	162.036	58.84			
HDPE/LDPE(40/60)-F	HDPE/LDPE	120.073	127.868	108.399			
HDPE/LDPE(40/60)-Pul	HDPE/LDPE	122.691	127.973	107.059			
HDPE/LDPE(60/40)-F	HDPE/LDPE	121.095	130.199	129.545			
HDPE/LDPE(60/40)-Pul	HDPE/LDPE	122.997	128.321	105.658			

Referring to Figures 12A,B-18A,B, DSC thermograms for different as-received flake feedstock and pulverized powder are shown. Before measuring the thermogram, the samples are heated to above their melting temperature and then cooled to ambient temperature at 10°C/minute. A striking difference is seen in Figures 12A,B-13A,B for as-received PP flake and pulverized PP powder. In particular, there is a large difference in the melting peaks of as-received PP flakes and pulverized PP powder as shown in Figures 12A,B. Despite identical thermal histories, the pulverized sample had two or possibly three distinct melting peaks (Figures 12A,B) not observed in the as-received PP flake sample. The crystallization peak is shifted to lower temperature for a pulverized material as shown in Figures 13A,B. There is observed a change in the crystalline PP phase after pulverization as shown by a 4 degree C decrease in the onset of Tc and a much sharper peak.

Figures 14A,B are thermograms for a 70%HDPE/30%PP blend of as-received flake feedstock and pulverized powder showing crystallization peaks. The ΔHc of the pulverized sample is 60% larger than that of the as-received sample.

Surprisingly, the largest degree of difference in thermal behavior was observed with as-received 60%HDPE/40%LDPE feedstock flake blends and similar pulverized blends. It is known that these polyolefins are incompatible because of their different densities. Referring to Figures 15A,B, a double melting peak observed for the as-received sample is changed into a single, narrower peak for the pulverized sample. After heating to the melt state and cooling to ambient temperature at 10°C/minute, a similar narrowing of the melting peak is evident from the as-received sample to the pulverized sample, Figures 16A,B. This shows that permanent change has occurred and that the DSC is not

detecting temporary mixing effects caused by the extruder. Figures 17A,B show the crystallization isotherm to be unified and sharpened by solid state shear pulverization pursuant to the invention. The same results were also observed for a 40%HDPE/60%LDPE blend. The data provide strong indication that the solid state shear pulverization of the scrap flake material imparts a high degree of in-situ compatibilization to the polymer components involved.

Figures 18A,B are a thermogram showing melting traces for a ternary blend of 60%HDPE/30%LDPE/10%PP as-received and pulverized. Similar thermogram results as described above are evident in Figures 18A,B.

The above-described embodiment of the present invention is advantageous in that comminuted (e.g. flake) scrap material can be solid state pulverized to particulates (e.g. powder) that are directly usable as powder feedstock in conventional melt processing techniques, such as rotational molding, blow molding, extrusion, spray coating and others requiring powder feedstock. Moreover, commingled, unsorted plastic scrap can be recycled without the need for costly sortation and in a manner to achieve in-situ compatibilization of different polymers present in the scrap in a once-through pulverization operation to produce recycled, polymeric particulates. Furthermore, the need for compatibilizing and/or reinforcing agent additions in the event two or more thermodynamically incompatible polymers are present in the scrap is avoided, thereby reducing the cost of recycling. This embodiment also is advantageous in that sorted or unsorted, commingled, mixed-color plastic scrap can be recycled to produce recycled, polymeric particulates that are unexpectedly conventionally melt processable to substantially homogeneous light color without color streaking or marbleizing. High value, low cost recycled powder

products, as well as products molded or otherwise formed of the powder, from sorted or unsorted, commingled multi-colored polymeric scrap material thus can be provided, increasing utilization of available plastic scrap.

Although the embodiment of the invention described in detail hereabove relates to the recycling of sorted or unsorted post-consumer and/or post-industrial polymeric scrap material, the invention is not so limited. For example, the invention also can be practiced to solid state pulverize feedstock comprising mixtures of post-consumer and/or post-industrial polymeric scrap and virgin polymeric material feedstock comprising one or more virgin polymeric materials.

In solid state pulverizing mixtures of one or more scrap polymeric materials and one or more virgin polymeric material, the weight %'s of the scrap and virgin materials can be varied over wide ranges to suit particular needs and material availability. For purposes of illustration and not limitation, a mixture of 75 weight % of virgin LDPE (translucent white color pellets) and 25 weight % of the aforementioned chopped scrap LDPE flakes (multi-color) was made and solid state pulverized pursuant to the invention using the aforementioned Berstorff ZE-40A twin screw extruder. The 75/25 virgin/flake LDPE mixture was solid state pulverized without heating (all barrel zones cooled) using processing parameters similar to those set forth in TABLE I for "No heat" solid state pulverization of a solely scrap flake LDPE feedstock.

The solid state pulverized particulates (powder) were injection molded in the manner described hereabove for the pulverized scrap particulates. The initial 75/25 virgin/flake LDPE feedstock also was injection molded for comparison purposes.

The physical properties of the injection molded specimens are shown in TABLE IV below. It can be seen that the physical properties of the injection molded pulverized powder specimens are generally comparable to those exhibited by the injection molded 75/25 virgin/flake LDPE specimens.

The injection molded pulverized powder LDPE specimens exhibited a translucent white color. The injection molded virgin/flake LDPE specimens exhibited a light peach color.

Moreover, a mixture of 60 weight % virgin LDPE (pellets) and 40 weight % chopped scrap LDPE flakes was made and solid state pulverized using the Berstorff ZE-40A twin screw extruder with barrel heating (barrel zones 2-4 heated) using processing parameters similar to those set forth in TABLE I for "w/heat" solid state pulverization of a solely scrap flake LDPE feedstock.

The physical properties of injection molded pulverized powder specimens and injection molded 60/40 virgin/flake specimens also are shown in TABLE IV below. It can be seen that the physical properties of injection molded pulverized powder specimens are generally comparable to those exhibited by the injection molded 60/40 virgin/flake specimens.

The injection molded pulverized powder LDPE specimens exhibited a translucent white color. The injection molded virgin/flake LDPE specimens exhibited a medium peach color.

TABLE IV

MATERIALS	TENSILE PROPERTIES			NOTCHED IZOD IMPACT	HDT DEG. C 66 PSI	HARDNESS SHORE D
	Yield PSI	Ultimate PSI	% Elong			
LDPE-v/LDPE-F	1450		120	NA	NA	48
75/25 Pulv No heat	1440		130	NA	NA	50
LDPE-V/LDPE-F	1420		135	NA	NA	51
60/40 Pulv Heat	1440		150	NA	NA	51

V = Virgin Pulv = Pulverized

F = Flake

NA -Not applicable (Samples were too flexible for impact and HDT testing)

As mentioned, the present invention also envisions solid state pulverizing of one or more virgin polymeric materials as feedstock. If two or more thermodynamically incompatible virgin polymers are present in the virgin material feedstock, in-situ compatibilization of the virgin polymers should be achievable.

For purposes of illustration and not limitation, virgin LDPE pellets were solid state pulverized using the aforementioned Berstorff ZE-40A twin screw extruder with heating (barrel zones 2-4 heated) and without heating (all barrel zones cooled) pursuant to the invention.

The virgin LDPE pellets were solid state pulverized with heating (barrel zones 2-4 heated) using processing parameters similar to those set forth in TABLE I for "w/heat" solid state pulverization of the solely scrap flake LDPE feedstock. The virgin LDPE pellets were also solid state pulverized without heating (all barrel zones cooled) using processing parameters similar to those set forth in TABLE I for "No heat" solid state pulverization of scrap LDPE feedstock.

The solid state pulverized virgin LDPE particulates (powder) were injection molded in the manner described hereabove for the pulverized scrap particulates. The virgin LDPE pellets were similarly injection molded.

The physical properties of the injection molded specimens are shown in TABLE V below. It can be seen that the physical properties of injection molded pulverized specimens are generally comparable to those of injection molded pellet specimens.

Virgin PC pellets also were solid state pulverized with heating (barrel zones 2-4 heated) using the Berstorff ZE-40A twin screw extruder. The virgin PC pellets were solid state pulverized using processing parameters similar to those set forth in TABLE I for "W/heat" solid state pulverization of scrap PP thickness (slightly higher temperature).

The pulverized particulates and virgin pellets were injection molded as described hereabove. Thy physical properties of the injection molded specimens are shown in TABLE V. It can be seen that injection molded pulverized powder PC specimens and injection molded pellet specimens exhibited comparable physical properties.

TABLE V

MATERIALS	TENSILE PROPERTIES			NOTCHED IZOD IMPACT	HDT DEG. C 66 PSI	HARDNESS SHORE D
	Yield PSI	Ultimate PSI	% Elong			
LDPE-V (pellets)	2120		170	NA	NA	49
LDPE w/heat pulv	2030		160	NA	NA	52
LDPE pulv no heat	2040		140	NA	NA	50
PC-V (pellets)	8850		105	13.8	140	83
PC-V pulv w/heat	8920		100	11.2	140	84

LDPE-V = Virgin resin NA 355, Quantum Chemical Co.
PC-V = Virgin resin 301-22, Dow Chemical Co.
NA – Not applicable (Samples were too flexible for impact and HDT testing)

Regardless of the composition of the polymeric feedstock supplied to the extruder, the present invention is advantageous in that energy consumption of the solid state pulverization process is lower than that of conventional batch grinding processes. In addition, the present invention provides a continuous, once-through solid state pulverization process in contrast to conventional batch grinding techniques.

While the invention has been described in terms of specific embodiments thereof, it is not intended to be limited thereto but rather only to the extent set forth hereafter in the following claims.

The embodiments of the invention in which an exclusive property or privilege is claimed are defined as follows:

1. A method of making polymeric particulates, comprising:

a) supplying polymeric material having different colors to screw means of an extruder, and

b) rotating the screw means to transport said polymeric material along the length thereof and solid state pulverize said polymeric material to particulates that are melt processable to a substantially homogeneous color appearance without color streaking.

2. The method of Claim 1 wherein the polymeric material includes at least one of scrap polymeric particulates, virgin polymeric particulates and mixtures thereof.

3. The method of Claim 1 wherein the polymeric material comprises a plurality of polymeric materials having different compositions.

4. The method of Claim 1 wherein the polymeric material is solid state pulverized with only frictional heating thereof by engagement with said screws.

5. A method of making polymeric particulates, comprising:

a) supplying polymeric materials comprising two or more thermodynamically incompatible polymers to screw means of an extruder, and

b) rotating the screw means to transport said scrap material along the length thereof and solid state pulverize said material with in-situ polymer

compatibilization to produce particulates without the need for a compatibilizing agent.

6. The method of Claim 5 wherein the polymeric material is selected from at least one of scrap polymeric particulates, virgin polymeric particulates and mixtures thereof.

7. The method of Claim 5 wherein the polymeric material includes particulates having different colors.

8. The method of Claim 5 wherein the polymeric material further includes thermosetting scrap material.

9. The method of Claim 5 wherein said in-situ polymer compatibilization is evidenced by said pulverized particulates exhibiting a different thermogram from the unpulverized polymeric material of the same composition.

10. The method of Claim 5 wherein the pulverized particulates exhibit at least one of a different melting peak and crystallization peak from the unpulverized material.

11. The method of Claim 5 wherein the polymeric material is solid state pulverized with only frictional heating thereof by engagement with said screws.

12. A method of making polymeric particulates, comprising:

a) supplying polymeric material to screw means of an extruder, and

b) rotating the screw means to transport said polymeric material along the length thereof to solid

state pulverize said polymeric material without melting to pulverized particulates.

13. A method of making polymeric particulates, comprising:

a) supplying polymeric material to screw means of an extruder, and

b) rotating the screw means to transport said polymeric material along the length thereof with only frictional heating by engagement with said screw means to solid state pulverize said polymeric material to pulverized particulates.

14. A method of making recycled polymeric particulates, comprising:

a) supplying comminuted polymeric scrap material to screw means of an extruder, and

b) rotating the screw means to transport said scrap material along the length thereof and solid state pulverize said scrap material to particulates that are directly melt processable to shape by melt processing techniques using powder feedstock.

15. The method of Claim 14 wherein polymeric scrap material is supplied to said screws as scrap flakes.

16. The method of Claim 14 wherein polymeric scrap material including an amount of virgin polymeric material mixed therewith is supplied to the screw means.

17. The method of Claim 14 wherein the flake scrap material is solid state pulverized with only frictional heating thereof by engagement with said screws.

18. A method of making an article of manufacture having a substantially homogenous color from multi-colored polymeric material, comprising:

a) supplying multi-colored polymeric material to screw means of an extruder,

b) rotating the screw means to transport said polymeric material along the length thereof and solid state pulverize said polymeric material to particulates, and

c) melt processing said pulverized particulates to a substantially homogeneously colored article of manufacture characterized by the absence of color streaking.

19. The method of Claim 18 wherein said pulverized particulates are directly melt processed by melt processing techniques using a powder feedstock.

20. A method of making an article of manufacture, comprising:

a) supplying polymeric materials including two or more thermodynamically incompatible polymers to screw means of an extruder,

b) rotating the screw means to transport said polymeric material along the length thereof and solid state pulverize said polymeric material with in-situ polymer compatibilization to produce particulates, and

c) melt processing said pulverized particulates to form an article of manufacture.

21. A method of making an article of manufacture from comminuted polymeric scrap material, comprising:

a) supplying comminuted polymeric scrap material to screw means of an extruder,

b) rotating the screw means to transport said scrap material along the length thereof and solid state pulverize said scrap material to particulates, and

c) subjecting said pulverized particulates as powder feedstock to melt processing to form an article of manufacture.

22. The method of Claim 21 wherein said pulverized particulates are supplied as powder feedstock for rotational molding, blow molding, extrusion or spray coating.

23. The method of Claim 21 wherein the comminuted scrap material includes different colored particulates of the same or different composition.

24. The method of Claim 23 wherein the scrap material includes an amount of virgin polymeric material.

25. Solid state pulverized polymeric scrap particulates produced from mixed-color polymeric material, said particulates being melt processable to a substantially homogeneous color without color streaking.

26. Solid state pulverized polymeric particulates produced from two or more thermodynamically incompatible polymers, said polymers being in-situ compatibilized by solid state pulverization as evidenced by said pulverized particulates exhibiting a different thermogram from unpulverized polymeric materials of the same composition.

27. Solid state pulverized polymeric particulates produced from two or more thermodynamically incompatible, multi-colored polymers commingled,

unsorted, multi-colored polymeric said polymers being in-situ compatibilized by solid state pulverization as evidenced by said pulverized particulates exhibiting a different thermogram from unpulverized material of the same composition, said particulates being substantially homogeneously colored despite being produced from mixed-color scrap material.

28. Recycled, solid state pulverized polymeric scrap particulates, said particulates being directly processable as powder feedstock by melt processing techniques requiring powder feedstock.

29. The particulates of Claim 25 wherein the particulates exhibit enhanced reactivity as compared to like unpulverized polymeric material as measured by electron spin resonance spectroscopy and differential scanning calorimetry.

30. The particulates of Claim 26 wherein the particulates exhibit enhanced reactivity as compared to like unpulverized polymeric material as measured by electron spin resonance spectroscopy and differential scanning calorimetry.

31. An article molded or extruded from the particulates of Claim 25.

32. An article molded or extruded from the particulates of Claim 26.

33. An article molded or extruded from the particulates of Claim 28.

34. A powder coating formed from the particulates of Claims 25.

35. A powder coating formed from the particulates of Claims 26.

36. A powder coating formed from the particulates of Claims 28.

37. Powder feedstock comprising pulverized particulates of Claims 25.

38. Powder feedstock comprising pulverized particulates of Claims 26.

39. Powder feedstock comprising pulverized particulates of Claims 28.

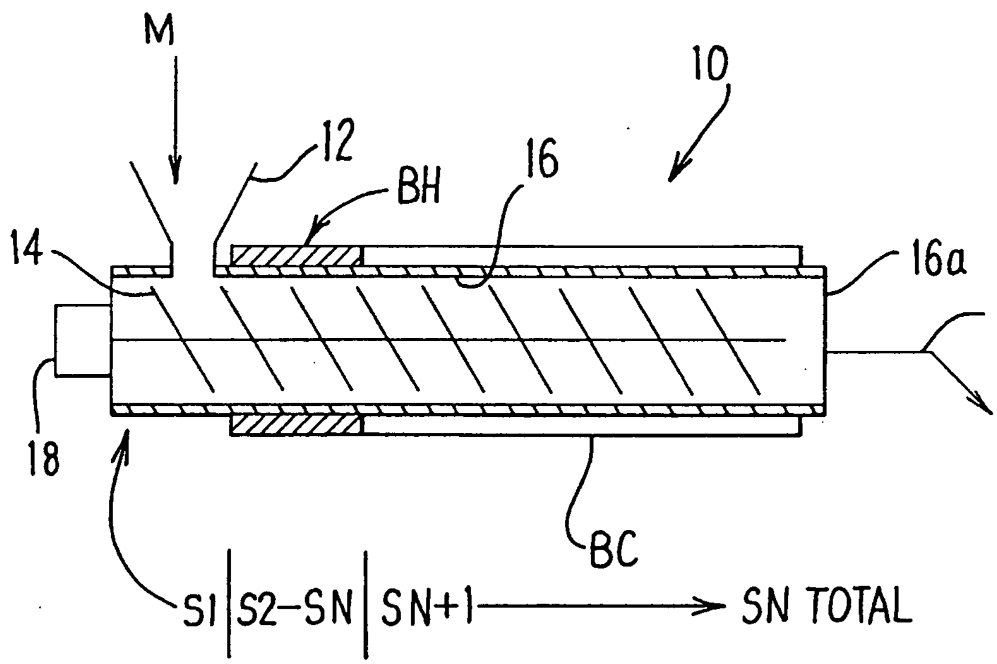


FIG. 1

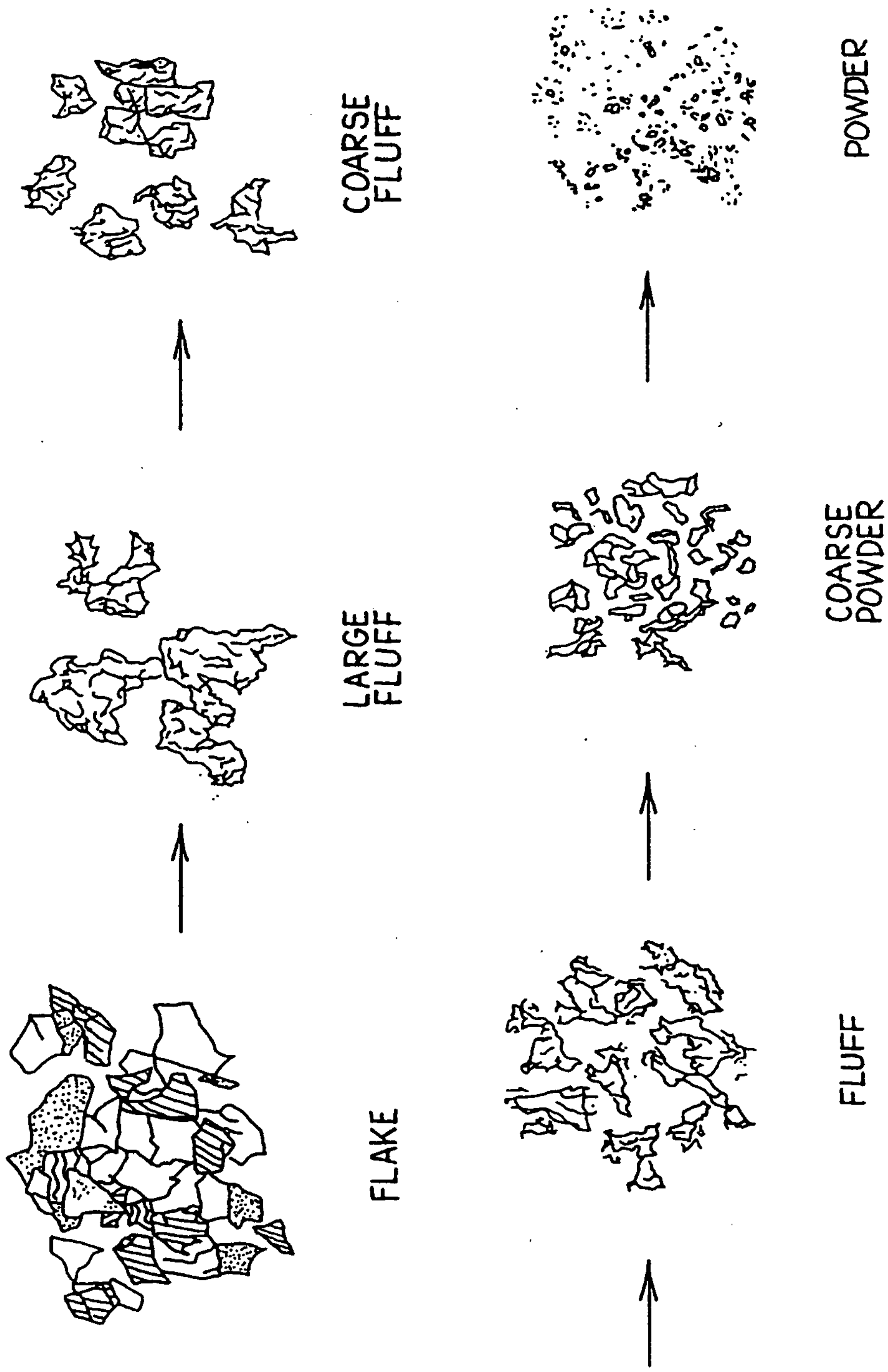
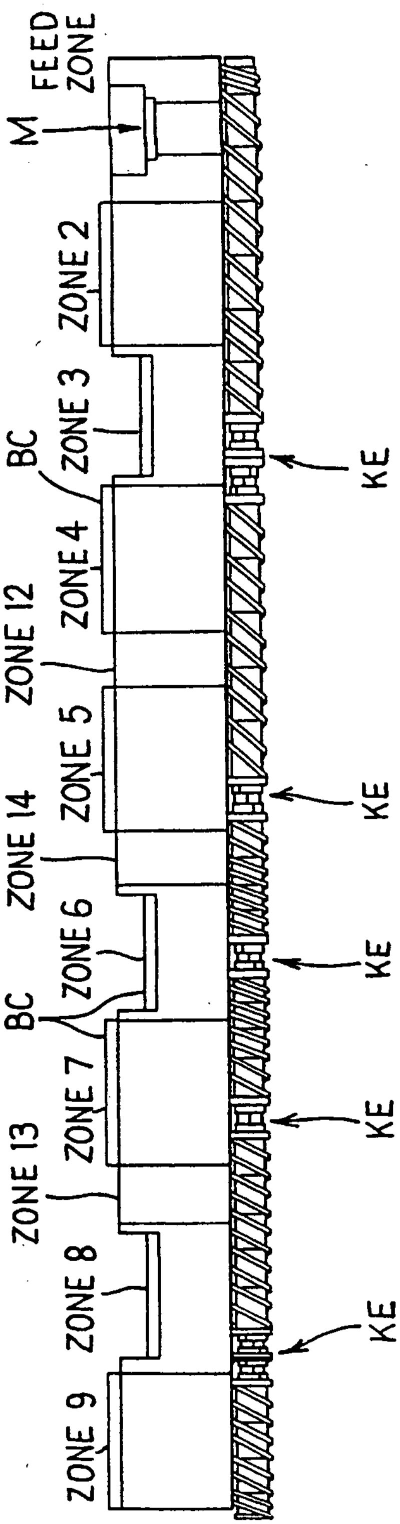


FIG. 2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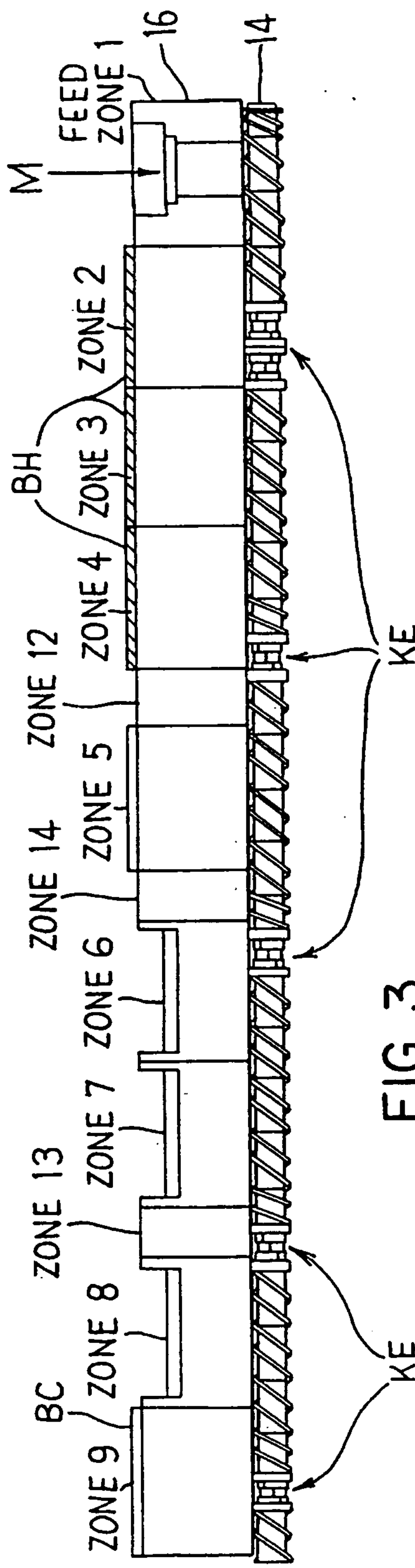
ZONE 2
ZONE 3
BC
ZONE 4
ZONE 12
ZONE 5
ZONE 14
ZONE 6
BC
ZONE 7
ZONE 13
ZONE 8
ZONE 9
KE
KE
KE
KE
KE

FIG. 4


FEED
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14
M
ZONE 2
BH
ZONE 3
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ZONE 8
BC
ZONE 9
KE
KE

FIG. 3

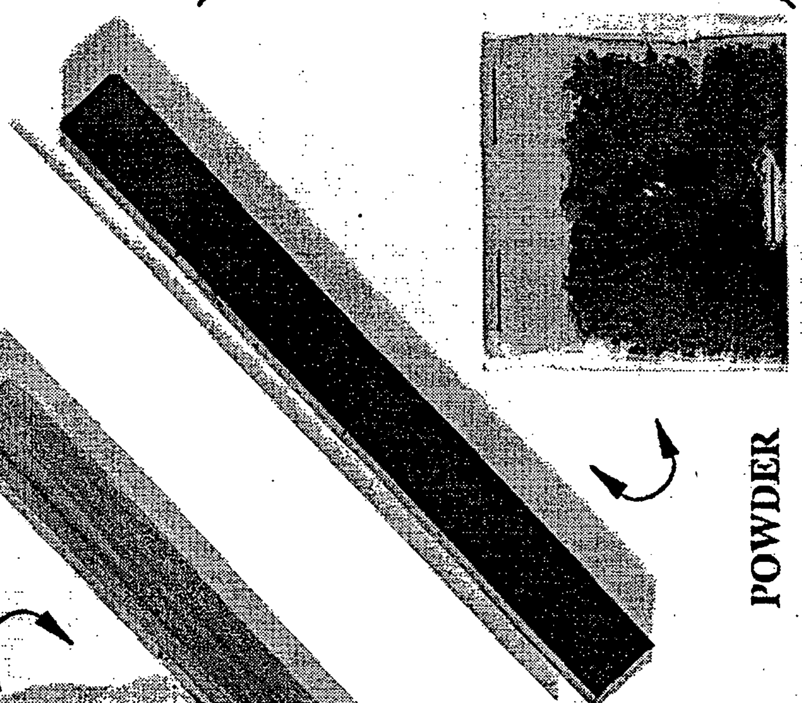


FIG. 5B

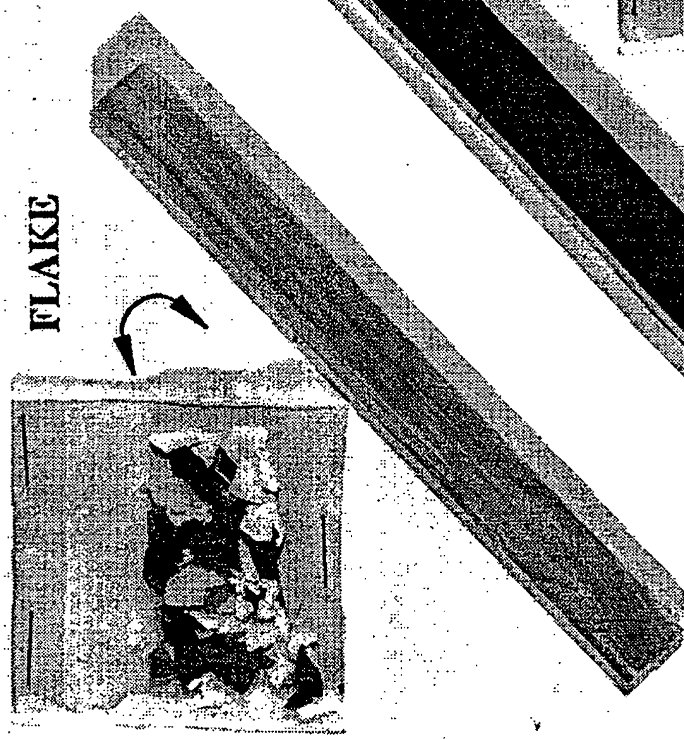
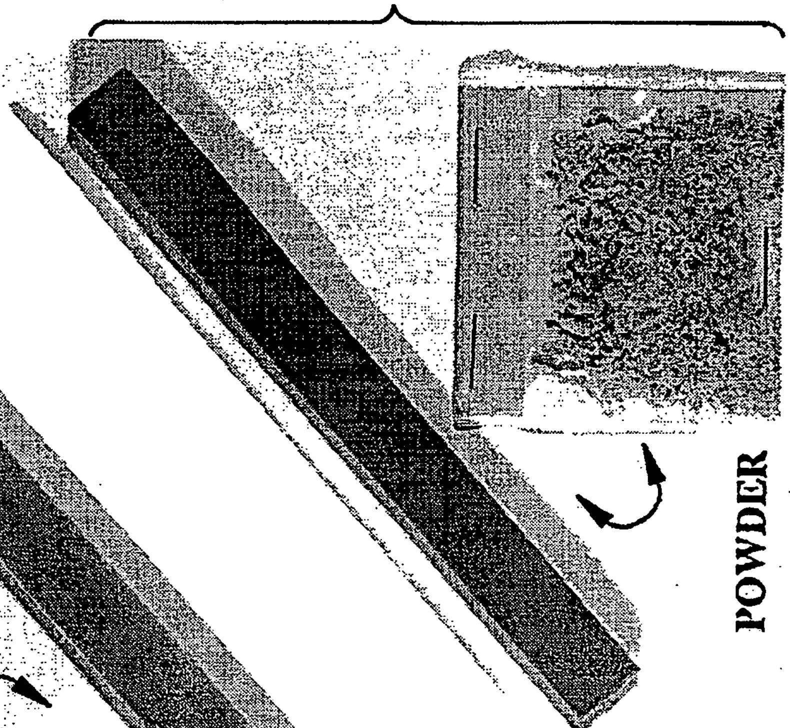


FIG. 5A

FIG. 6B



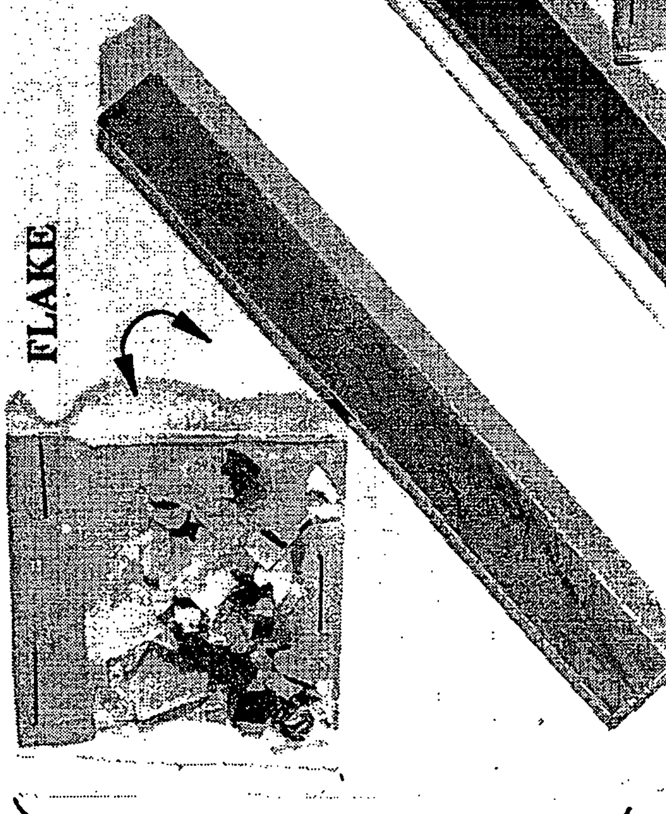


FIG. 6A

FIG. 7B

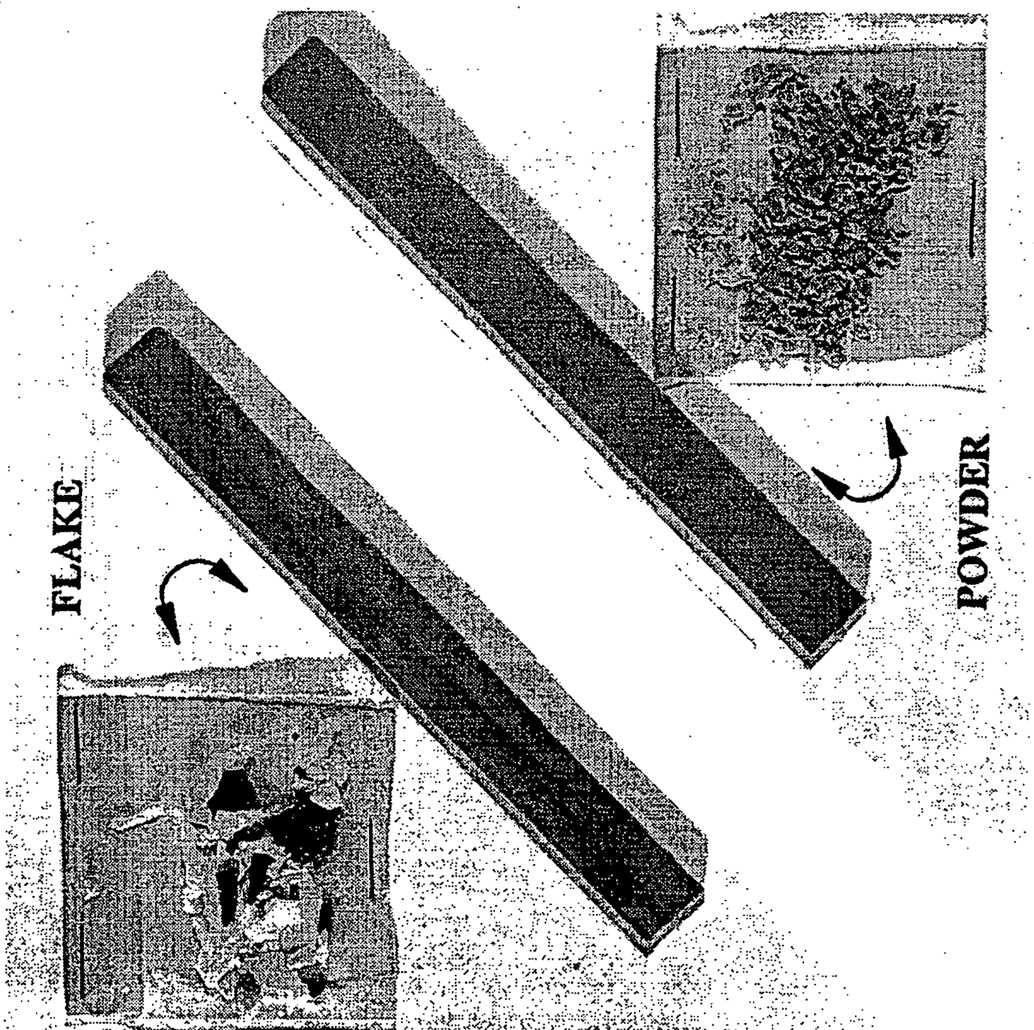


FIG. 7A

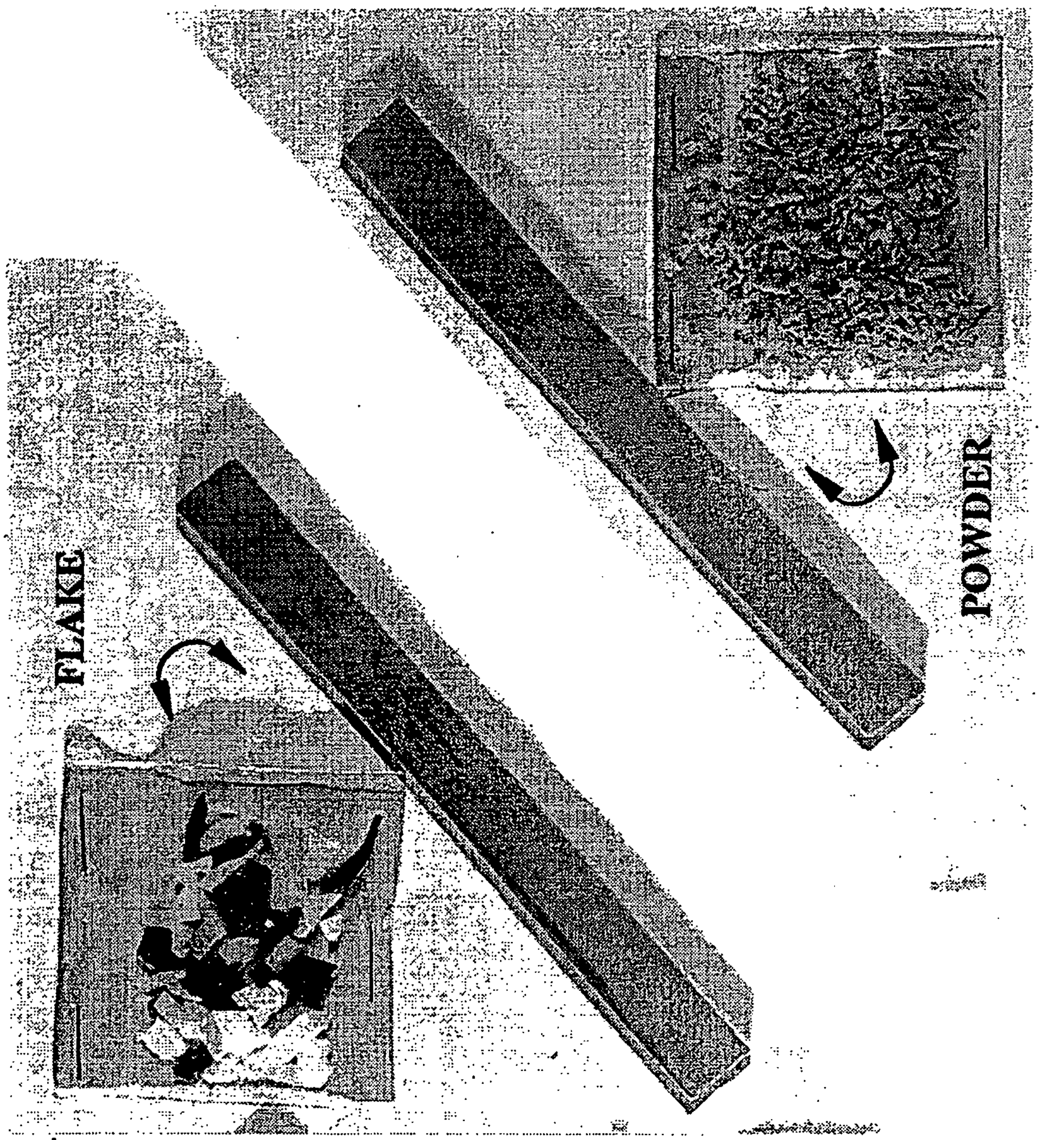
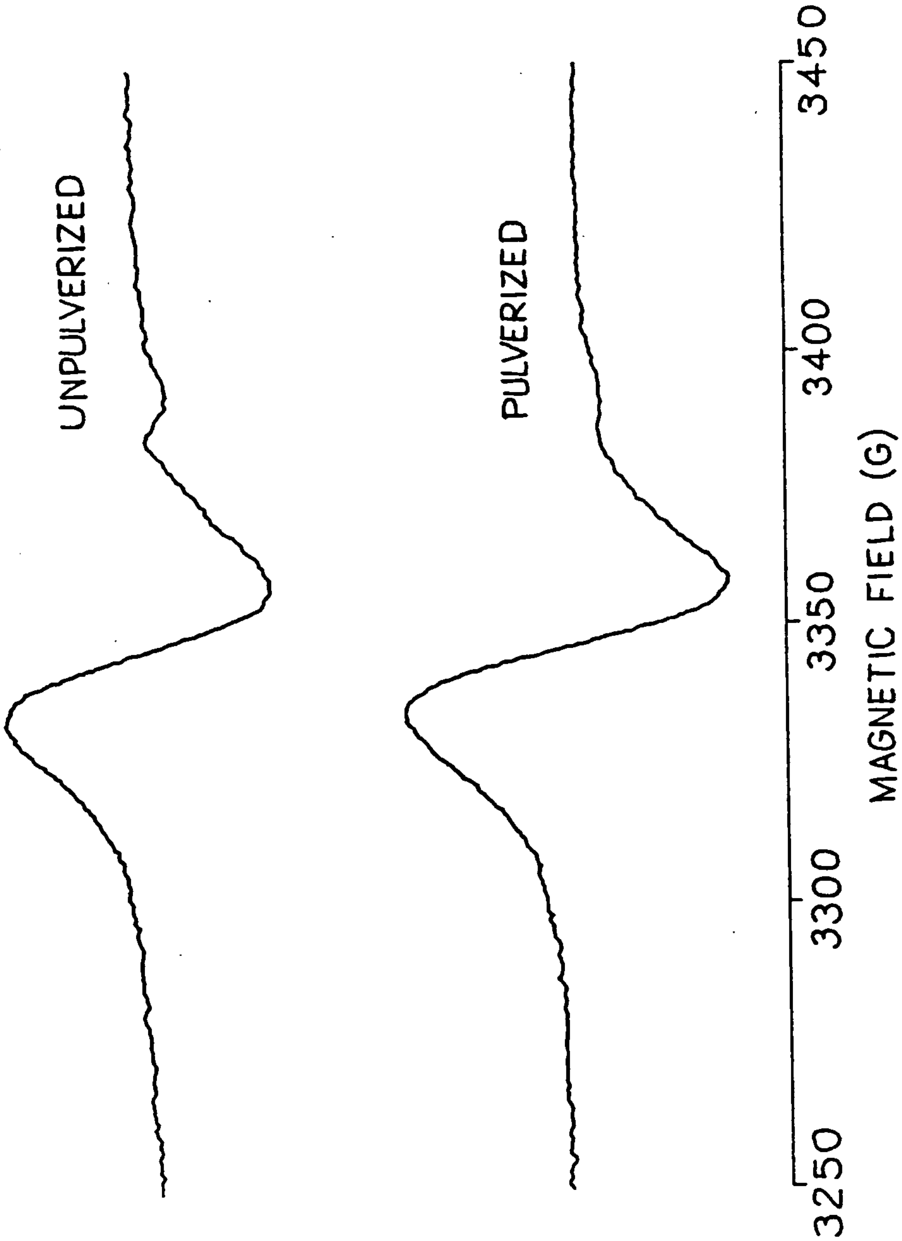


FIG. 8A

FIG. 8B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PULVERIZED
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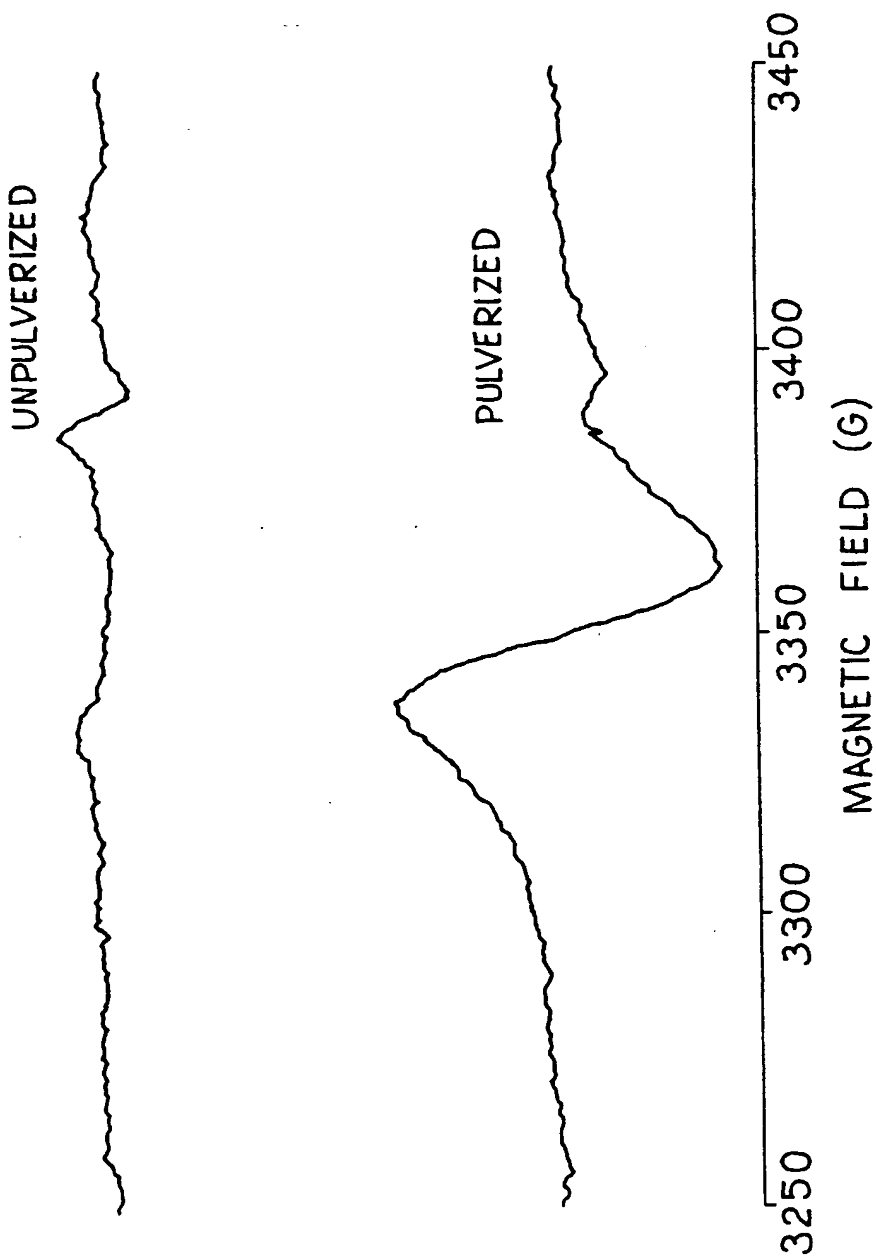
3300
3350
3400
3450
MAGNETIC FIELD (G)

FIG. 9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PULVERIZED
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3300
3350
3400
3450
MAGNETIC FIELD (G)

FIG. 10

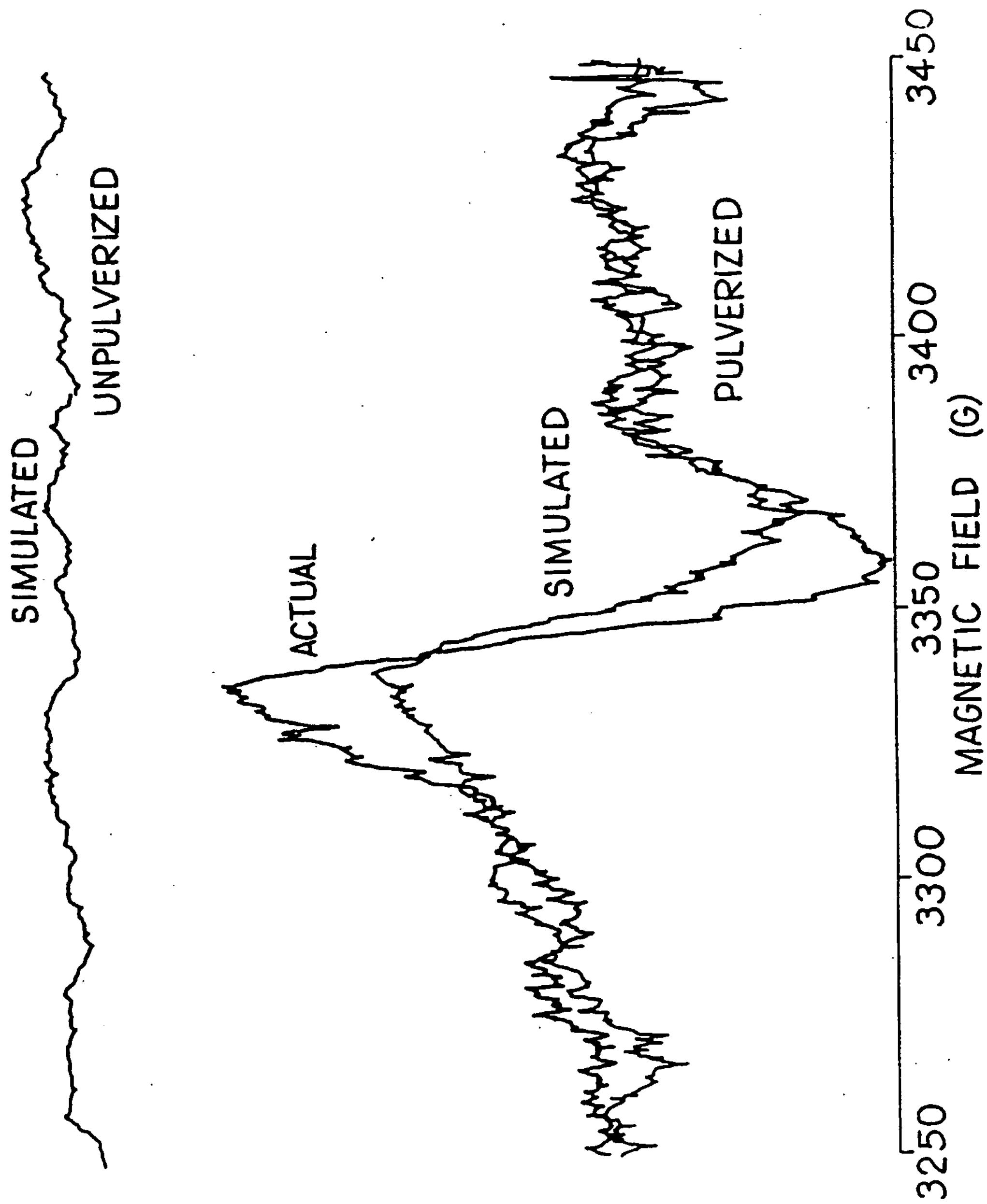

SIMULATED
UNPULVERIZED
ACTUAL
SIMULATED
PULVERIZED
3250
3300
3350
3400
3450
MAGNETIC FIELD (G)

FIG. 11

FIG. 12A

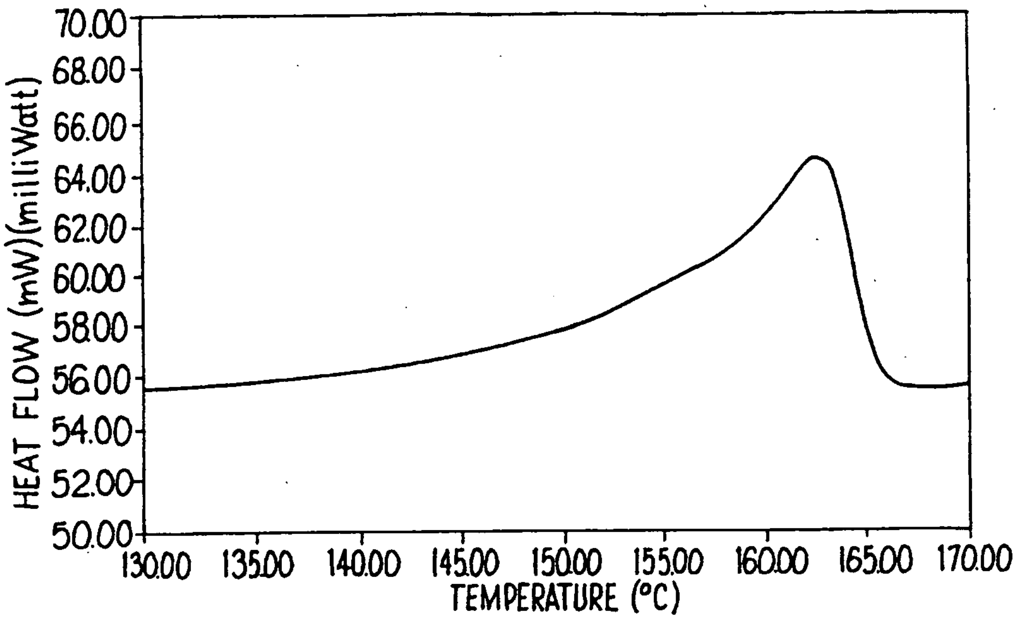


FIG. 12B

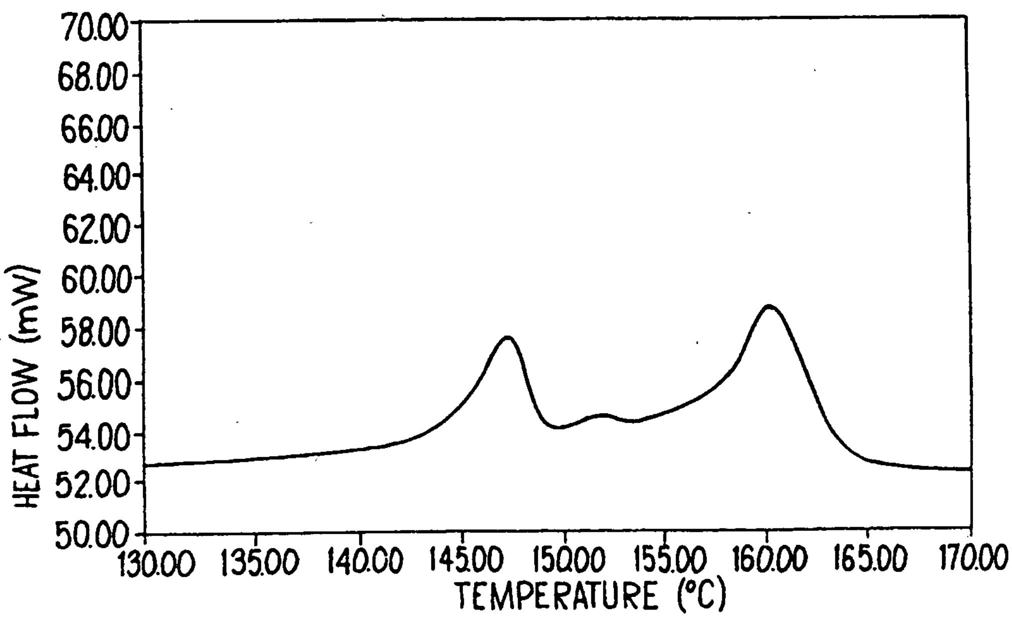


FIG. 13A 12/17

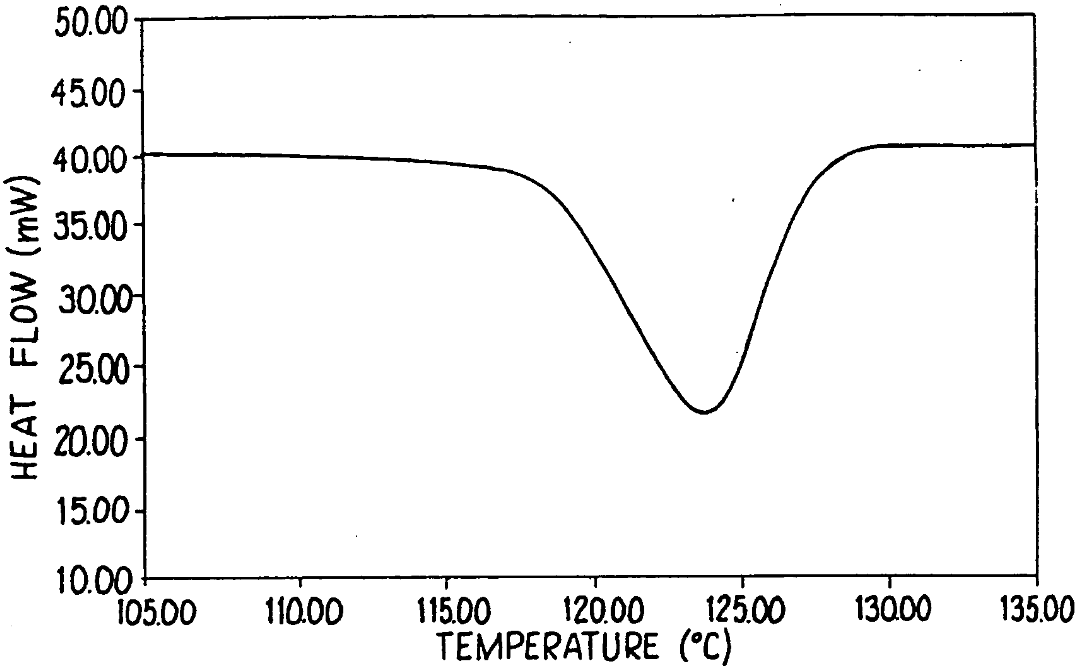


FIG. 13B

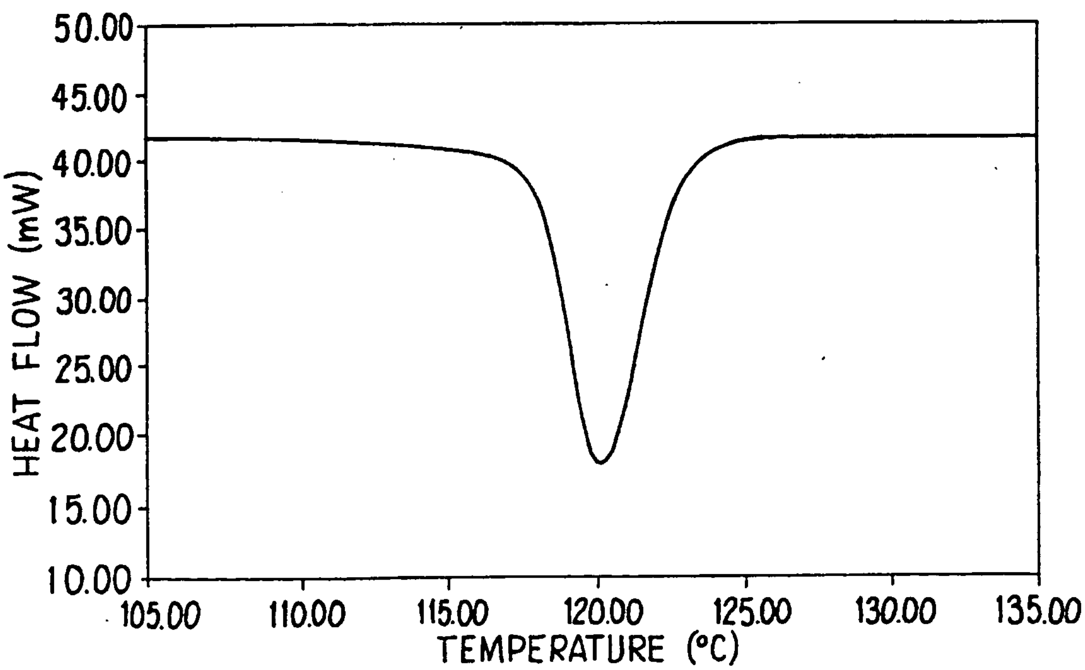


FIG. 14A

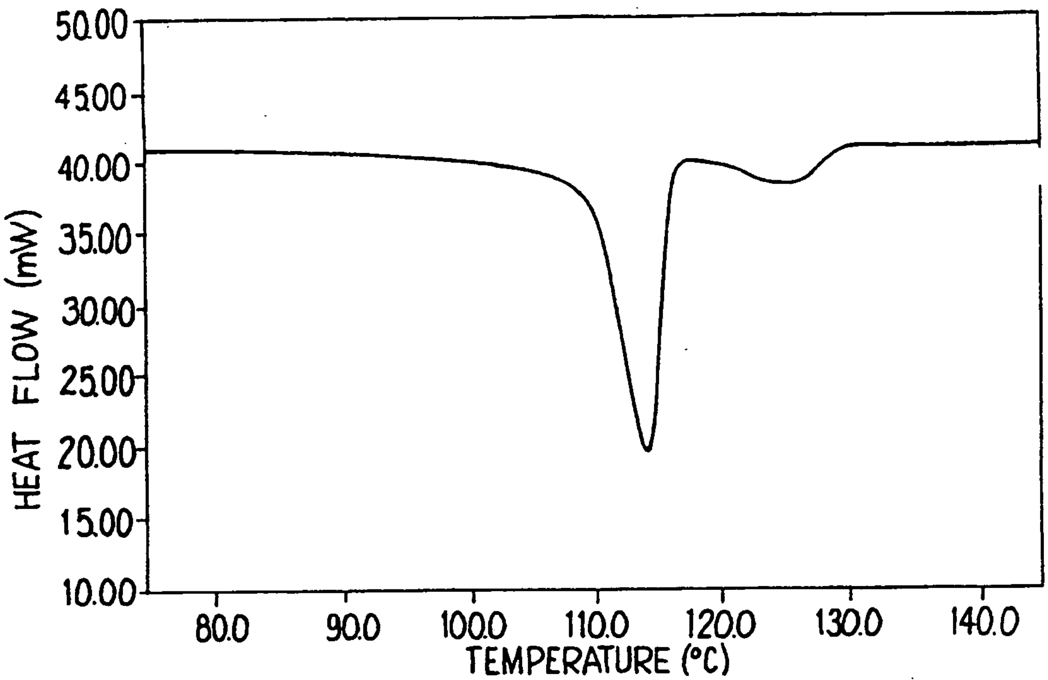


FIG. 14B

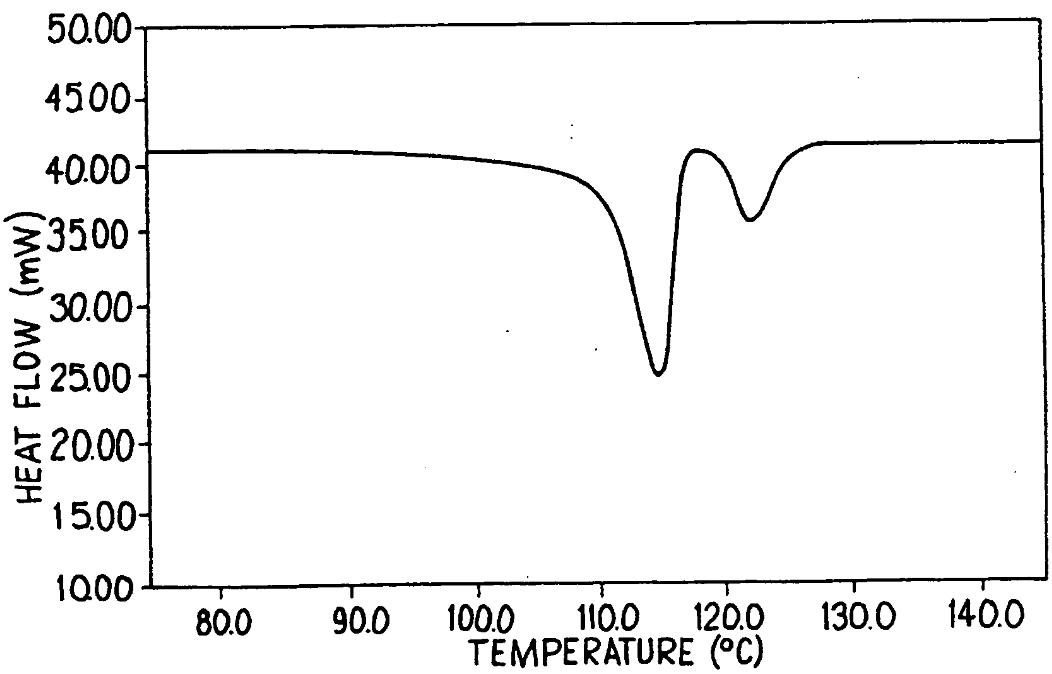


FIG. 15A

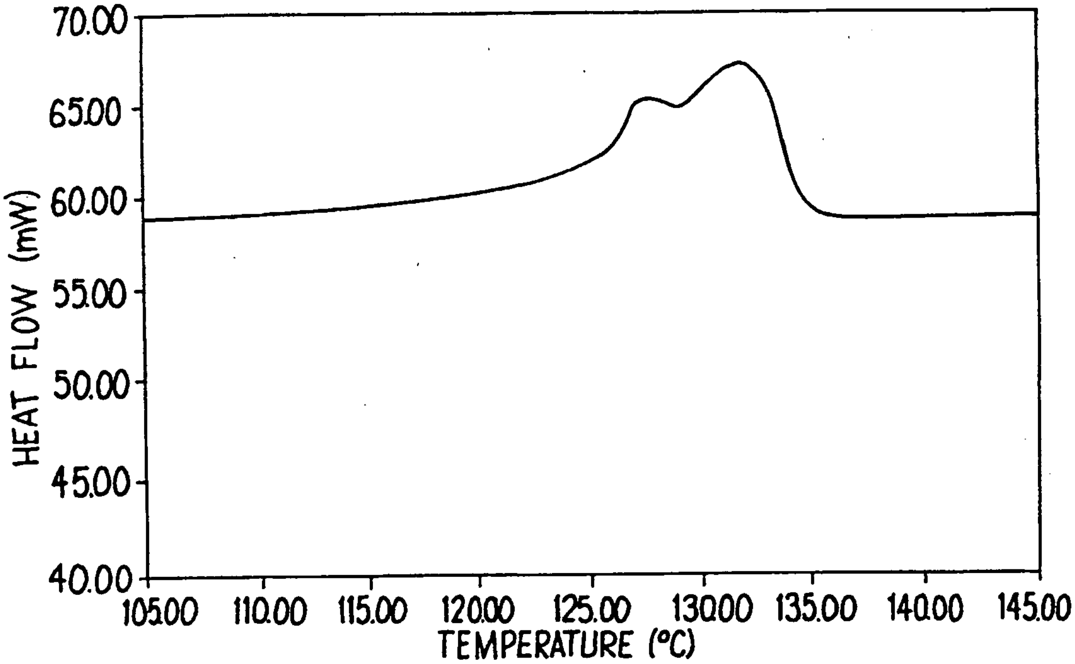


FIG. 15B

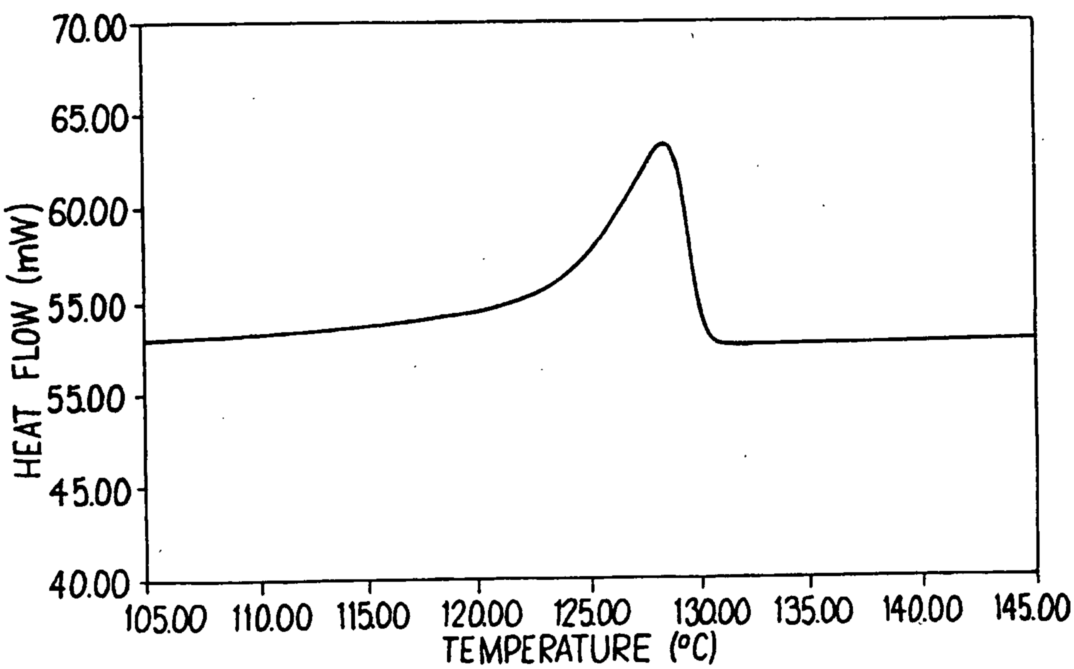


FIG. 16A

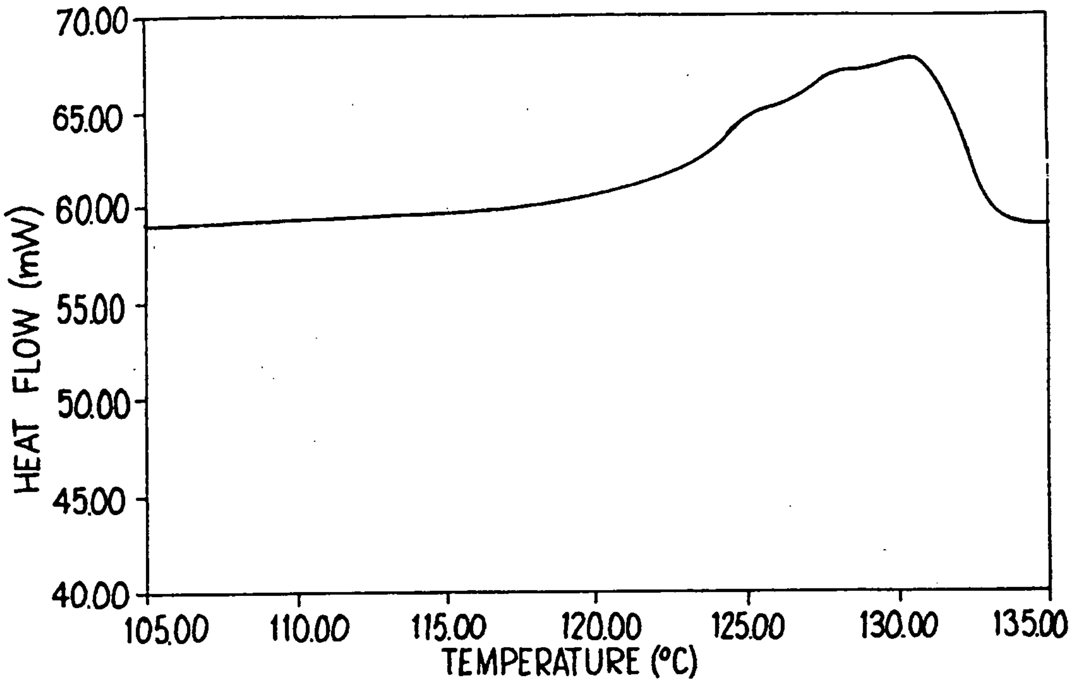


FIG. 16B

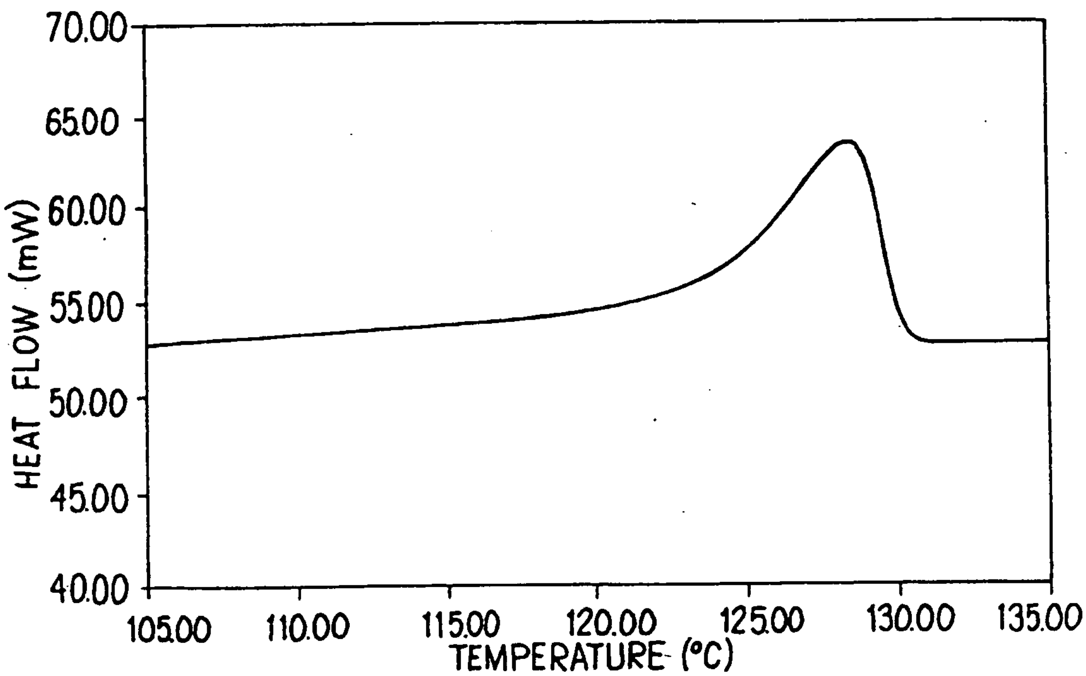


FIG. 17A

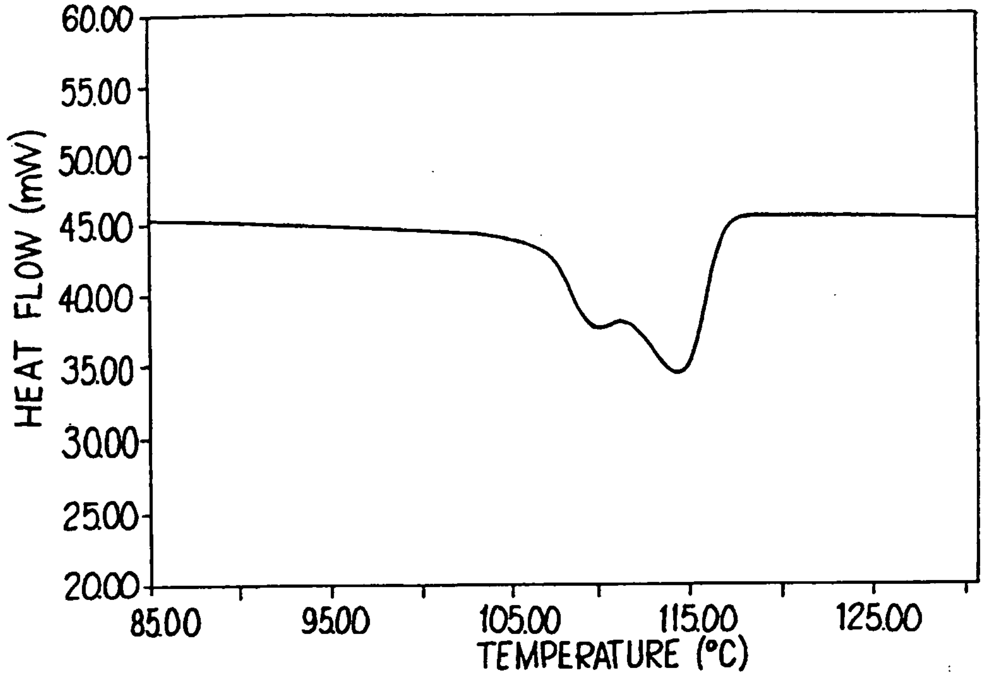


FIG. 17B

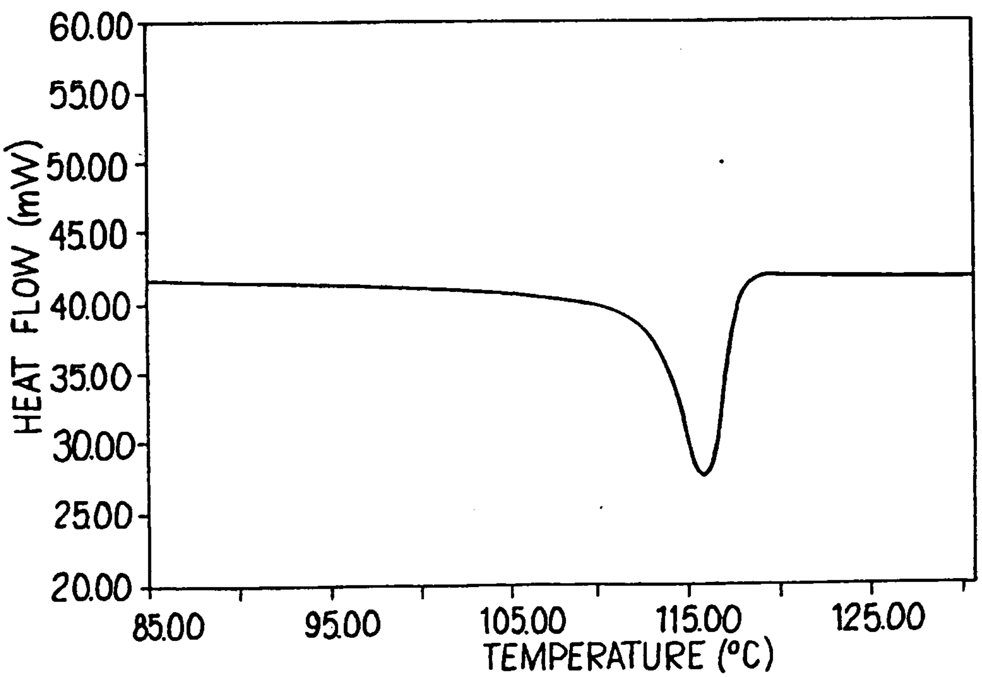


FIG. 18A

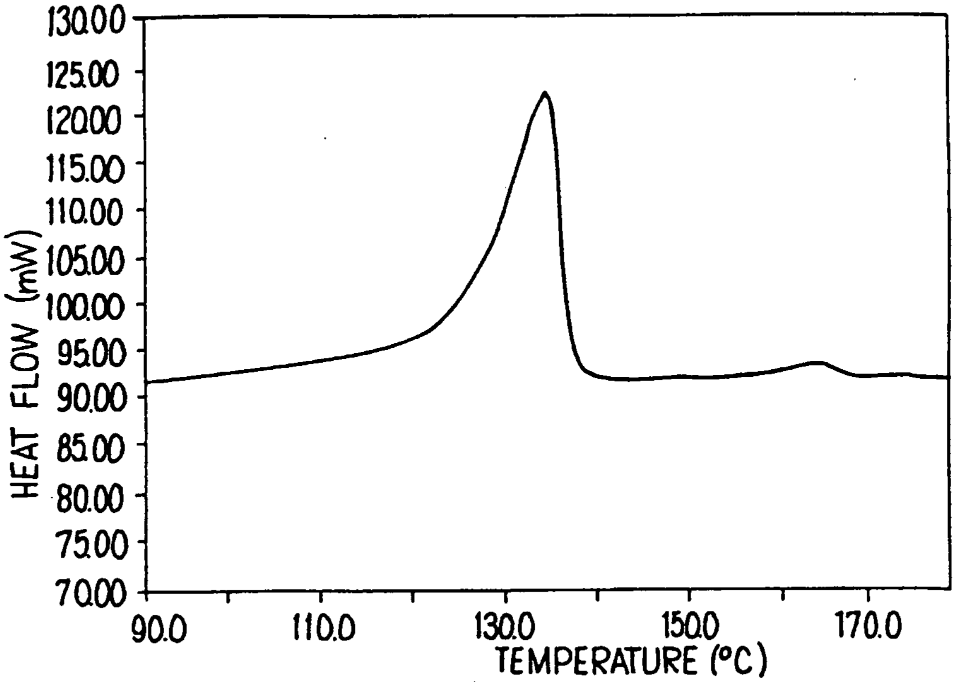
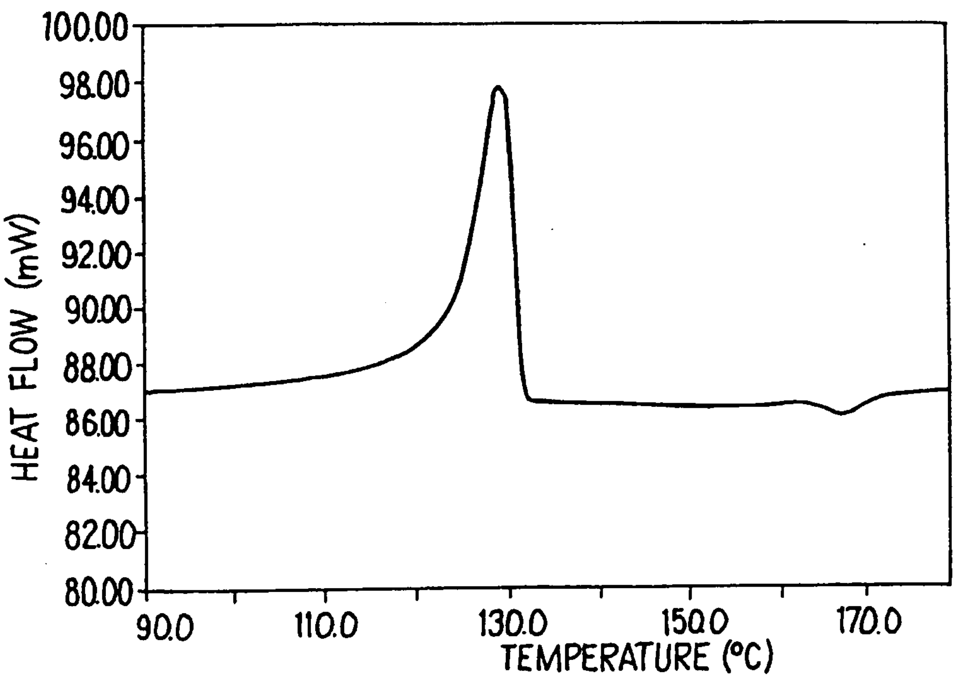


FIG. 18B



INTERNATIONAL SEARCH REPORT

International application No.
PCT/US94/13972

A. CLASSIFICATION OF SUBJECT MATTER

IPC(6) :B02C 19/12
US CL :241/23; 425/208; 525/71
According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 241/23; 425/208; 525/71

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US, A, 4,607,797 (ENIKOLOWPOW ET AL.) 26 August 1986, columns 2-4.	1-39
Y	US, A, 4,917,834 (HADERMANN ET AL.) 17 April 1990, col. 3, lines 1-30.	1-39
Y	US, A, 3,976,730 (CUSHING) 24 August 1976, col. 2, lines 1-44.	1-39

☐ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

* Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be part of particular relevance

"E" earlier document published on or after the international filing date

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"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
07 FEBRUARY 1995	10 APR 1995
Name and mailing address of the ISA/US Commissioner of Patents and Trademarks Box PCT Washington, D.C. 20231	Authorized officer PATRICK R. DELANEY
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